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INVESTIGATION OF SOLIDIFICATION OF NITROUS OXIDE UNDER FLOW CONDITIONS

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Air Force Weapons Laboratory
Kirtland Air Force Base, New Mexico

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AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
Kirtland Air Force Base, NM 87117

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An analysis and a test program were executed to determine if a problem existed when liquid N_2O was dumped from its tank, or gaseous N_2O was vented to the atmosphere. It appeared that the gas or liquid might solidify in the line and thus block further flow of the N_2O. Analysis and testing of the gas venting case showed that the gas could not solidify in the vent line under existing operating conditions. The gas venting process causes evaporation (and therefore, cooling) of the liquid N_2O in the tank and sufficient (OVER)

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ABSTRACT (Cont'd)

cooling can cause solidification of this liquid. Such solidification was observed during the gas vent tests.

Analysis of the liquid dumping showed that solidification of the liquid N_2O might occur if the pressure in the line fell below 12.7 psia. Flow experiments were run with the liquid N_2O dumping into a large vacuum chamber which simulated the high altitude environment during flight. The piping configuration used in the tests was identical to that of the airborne system. The tests showed that the pressure at the dump line exit remained above 12.7 psia for all conditions tested; therefore, no solidification occurred in the lines. The N_2O transformed to snow after it exited from the dump line and entered the vacuum chamber. This is in accord with the analysis and presents no operational problems for the dump system.

CONTENTS

SECTION I

THE PROBLEM

Under emergency conditions, it may be necessary to land the Airborne Laser Laboratory (ALL) aircraft with the propellant tanks empty. For this reason, the design provides for dumping liquid propellants and venting gaseous propellants to the atmosphere. Indications were that dumping and venting the N_2O tank might be problematic. Two situations occurred in which liquid N_2O was dumped in the atmosphere and promptly formed a solid. One of these incidents was at the National Bureau of Standards Laboratory in Boulder, Colorado, while the other took place at Edwards AFB, California. These incidents generated concern regarding possible solidification of the N_2O in the dump and/or vent lines if the tank were emptied during flight. It was felt that if solidification took place in the lines, the lines might be blocked and the N_2O tank could not be emptied. This potential problem required investigation.

The remainder of this report describes both the theoretical and experimental work done in order to evaluate the problem. The theoretical analysis aided greatly in understanding what was happening and why, but it was incapable of assessing whether or not an operational problem existed during dumping of the N_2O. This question was finally settled by a test program, and it was found that dumping and venting N_2O presented no operational problems. When reading this report one should keep in mind that the primary function of the test program was to assess a possible operational problem, and not to acquire fundamental knowledge about the properties of N_2O.

SECTION II

THEORETICAL ANALYSIS

The analyses of the dumping and venting processes can best be done with the aid of a schematic diagram of the N_2O system, together with the thermodynamic properties of the N_2O. Figure 1 shows that the N_2O dump and vent systems are not complicated. Under normal operating conditions, the N_2O in the tank is maintained at 0°F by the cooling coils shown in the figure. At this temperature, the vapor pressure of the N_2O in the ullage volume is 279 psia. Gaseous helium is then added to the tank until the total tank pressure is 1200 psia. In order to dump liquid, the valve in the liquid dump line must simply be opened; gas venting requires opening the gas vent valve. The N_2O discharges to the atmosphere, and if the system is airborne the atmospheric pressure is, of course, lower than that at sea level. In this study, we are interested in four cases. These are (1) dumping the liquid with only N_2O vapor in the ullage volume; (2) dumping the liquid when the tank contains both N_2O and helium pressurant; (3) venting the tank when it contains N_2O only; and (4) venting the tank with helium pressurization.

The dumping and venting processes can be followed on the pressure enthalpy diagram for N_2O shown in figure 2. It is easier to follow an idealized process on the diagram than it is to follow the real process; therefore, the process is idealized by assuming that the dump and vent processes are adiabatic and steady state. The adiabatic assumption is very good since the liquid or gas is in the line for only a short period of time during dumping or venting, and there is little time for heat transfer. The steady state assumption is good for dumping liquid under helium pressure, but the assumption is less accurate for the case of dumping without helium and for both venting cases. In the cases being considered, the temperature and pressure of the N_2O in the tank vary somewhat with time. We will begin by considering the idealized processes, which occur at constant enthalpy. Deviations from ideality will be taken into account subsequently.

LIQUID DUMPING WITHOUT HELIUM

The first case that we will consider is liquid dumping when there is no helium pressurant in the N_2O tank. The tank is maintained at 0°F by a cooling system.

At this temperature, the saturated N_2O has a vapor pressure of 279 psia. The state of the saturated liquid in the tank is shown as state 1 in figure 2, while the corresponding saturated vapor is at state 3. If we assume that the flow is steady state adiabatic (constant enthalpy), the N_2O will follow path 1 → 2 beyond state 2, until the ambient pressure is reached. During the first part of this flow, vaporization will occur as the pressure in the vent line falls. This fall in pressure is caused by the pressure drop of the fluid flow. Vaporization will cause liquid temperature to decrease, and the two phase flow (liquid + vapor) will occur until state 2 is reached at some point in the vent line. This is shown schematically in figure 1. Just downstream of this point, the pressure falls below the triple point pressure, and the liquid in the line undergoes a change of phase:

LIQUID → SOLID + VAPOR

Thus, upstream of state 2 in figure 1 we have a liquid-vapor mixture while downstream we have a solid-vapor mixture. The fraction of vapor is greater just downstream of state 2 than just upstream of it. The presence of the solid N_2O may or may not obstruct the flow, depending upon whether or not the solid collects at turns, valves, etc. It should be noted that there is a pressure drop at the pipe outlet. If this pressure drop is sufficiently large, it will be possible to maintain the pressure above 12.7 psia (in the vapor + liquid region) throughout the line length, although the ambient pressure may be less than 12.7 psia. In such a case, no solidification would occur in the line, but solid might be formed after the N_2O left the line.

IDEAL CASE VERSUS REAL CASE

The effect of deviation from the ideal process must now be considered. There will be heat transfer from the dump line to the N_2O. This heat transfer will tend to curve the path from state 1 to the right, and the path will intersect the line representing the triple point at a position to the right of state 2. Thus, there will be more vapor and less solid in the dump line than is predicted by the ideal process.

The idealized process (constant enthalpy) assumed steady state. In the real case, this does not occur. When dumping begins, the pressure of the N_2O in the ullage volume decreases. To compensate for this pressure loss, some of the liquid will vaporize and the rest of the liquid will thus be cooled. The heat transfer rate to the tank is lower than the rate required to maintain constant tank temperature, and the tank will cool as dumping and vaporization proceed. The N_2O in the tank will remain saturated during cooling, the state of the liquid in the tank will follow the saturated liquid line (figure 2) downward as vaporization and cooling occur. Similarly, the state of the vapor in the ullage volume will move downward on the saturated vapor line as cooling progresses. The actual path followed by a small quantity of liquid is represented schematically by the dotted line of figure 2. This small quantity of liquid exited the tank after the tank had cooled to -40°F. The liquid gains a small amount of heat as it traverses the dump line, causing an enthalpy increase. No solid appears until the pressure has fallen below 12.7 psia. Each small quantity of liquid leaving the tank will follow a path similar to the dotted path shown in figure 2; however, each mass increment will have a different starting point on the saturated liquid line. It is important to note that no solid forms in either the ideal or the actual process unless the pressure falls below the triple point pressure of 12.7 psia. In this important respect, the actual and ideal processes are the same. They differ in the ratio of solid to gas that is formed when the pressure falls below 12.7 psia.

DUMPING WITH HELIUM PRESSURANT

The case of liquid dumping using helium as a pressurant is a natural extension of the case just discussed. For this case, the liquid is at a pressure of 1200 psia and a temperature of 0°F. This is shown as state 4 in figure 2. It has been assumed that the 0°F isotherm is vertical in the liquid region of the pressure-enthalpy diagram. This is strictly true for an incompressible fluid, but it is therefore assumed that the compressibility of liquid N_2O is small (reasonable assumption). For the idealized process (steady state adiabatic) the N_2O follows path $4 \rightarrow 1 \rightarrow 2$ and then into the solid-vapor region. The N_2O in the first part of the line will be all liquid. This condition will prevail until the pressure in the vent line has fallen to 279 psia (state 1), where vaporization begins. This point is shown schematically as state 1 in figure 1. Vaporization will continue until the pressure has fallen to 12.7 psia (state 2). At this point the reaction

$$\text{LIQUID} \rightarrow \text{SOLID} + \text{VAPOR}$$

occurs, and the N_2O becomes a solid-vapor mixture. The point at which this reaction occurs is shown schematically as state 1 in figure 1. The effects of heat transfer and non-steady-state conditions are the same for this case as for the previous one.

GASEOUS VENTING

The last two cases to be considered are those of gaseous venting. Venting without helium will be discussed first. The gas in the ullage volume is at state 3 of figure 2. The gas will follow a path vertically downward from state 3, in the idealized process. It has a vapor pressure of 279 psia. The gas expands to a lower pressure during venting, and follows the idealized path shown. This path is always in the vapor region for the idealized process being considered; therefore, neither liquification not solidification occur. Next consider two deviations from the assumed ideal conditions. First, heat transfer will occur between the gas and the vent lines (the process is not adiabatic). The design ambient temperatures range from 40°F to 140°F, therefore the vent lines will be warmer than the gas. Heat transfer will be from the lines to the gas, and the enthalpy of the gas will increase as it flows through the vent lines. Thus, the real process will curve to the right, while the ideal process is vertical. The second deviation is the lack of a steady state process. As gaseous N_2O is vented, the gas pressure will diminish causing some of the liquid to vaporize. The state of the vapor in the tank will move downward on the saturated vapor line. The actual process followed by a small quantity of gas which left the tank at -60°F is shown by the X curve in figure 2. Both the actual and the ideal processes remain completely in the vapor region, therefore, no solidification or liquification is expected in the lines during venting. It should be noted that the cooling caused by vaporization of liquid N_2O in the tank can result in the freezing of this liquid, if the tank temperature falls below -131.4°F (triple point temperature).

The last case to be considered is gaseous venting when the ullage volume contains both He and N_2O. If we assume that Dalton's law holds for this gas mixture, the N_2O vapor will expand along approximately the same path as it did in the previous case. Thus, liquification and solidification problems do not appear for either case of gas venting.

SECTION III

TEST APPARATUS

The theoretical considerations show that solidification can occur in the lines during liquid dumping if the line pressure drops below 12.7 psia. The analysis does not tell us whether or not the pressure will actually fall below 12.7 psia, nor does it tell us whether or not the line will be blocked if solidification does occur. Thus, the analysis provides information concerning which parameters control the solidification problem, but the final evaluation of the problem requires a test program.

The requirements for the test apparatus can be readily determined. We need an N_2O tank that can be pressurized by helium and a larger vacuum tank into which the N_2O can be dumped or vented. The vacuum tank must be capable of simulating the altitudes at which the aircraft will fly. Joining these two tanks are the vent and dump lines. The configuration of these lines is critical to the test program since the pressure drop of the flowing N_2O depends upon the line configuration. If the pressure drop across the line exit is sufficiently great, the pressure in the line will never fall below 12.7 psia. On the other hand, if solidification does occur in the line, the configuration of the line will determine whether or not the solid will block the line. For these reasons, the dump and vent lines were fabricated as duplicates of the lines used in the airborne system.

A schematic diagram of the test facility is shown in figure 3. The various components were selected on the basis of availability as well as their being suited for the job. The helium source is simply a K bottle with a pressure regulator. The N_2O tank is an oversized gas sampling bottle. In the actual system, the N_2O temperature is kept at 0°F. For the testing, the N_2O temperature was reduced to 32°F before each run, by means of an iced water bath. The enthalpy difference between saturated liquid at 32°F and saturated liquid at 0°F is about 15 BTU/lb, which is approximately 15 percent of the heat of vaporization. For the saturated vapors, the difference is about 1 BTU/lb. These small discrepancies should not be significant to the test results, and iced water will be used to cool the oxidizer to 32°F.

The vacuum tank is a 350 cubic feet vacuum chamber, which was evacuated to a pressure of several torr by mechanical vacuum pumps. The running time for any particular test was limited by the rate of pressure rise in this tank. For example assume that the dump tank pressure at the start of a test is 50 torr (0.965 psi) and the test will be terminated when the pressure has increased to 10 psia. Calculations show that the pressure will be 10 psia when the dump tank contains 23.2 pounds of N_2O vapor. Thus, the duration of the test is limited to the time required for 23.2 pounds of N_2O to flow through the system. This mass occupies 0.413 cubic feet at the conditions in the N_2O bottle, and the N_2O bottle can handle this volume.

The instrumentation used during the tests is shown in figure 3. Viewing ports were provided at the ends of lines, and on the vacuum tank, to permit visual observation of the phases present in the flow stream. Pressures were measured in the N_2O tank and at the outlet of the appropriate line (dump or vent). Recall that if the pressure at the dump line exit is greater than 12.7 psia, the theory predicts no solidification in the line.

SECTION IV

TEST PROGRAM AND PROCEDURE

The test program consisted of seven runs; four of these tested liquid dumping, two tested gas venting, and the last was a combination of venting and dumping. The conditions under which these tests were run are birefly described below:

Run A - Twenty-four pounds of liquid were dumped. Helium was used as a pressurant during the entire process.

Run B - Same as Run A, but only 12 pounds of liquid N_2O were used.

Run C - Twenty-four pounds of liquid were dumped. No helium was used. The only pressurant was the N_2O vapor in the tank, at the vapor pressure.

Run D - Same as Run C, but only 12 pounds of liquid N_2O were used.

Run E - Twelve pounds of N_2O were placed in the tank. No helium was used. The vapor in the ullage volume was vented.

Run F - Twelve pounds of N_2O were placed in the tank. Helium was added until the total pressure was 1200 psia, after which the helium source was disconnected. The gas in the ullage volume was vented.

Run G - Twelve pounds of N_2O were placed in the tank without the helium pressurant. The vapor in the ullage volume was vented until the tank pressure was 200 psia, at which time the vent valve was closed and the dump valve was opened.

The procedure followed was straightforward. Before each run, the vacuum tank was evacuated, the iced water bath was prepared, and the N_2O tank was charged with 12 or 24 pounds of N_2O from a K bottle. The filled N_2O tank then remained in the iced water bath for at least 30 minutes in order to cool. If helium pressure was required, the helium valve was opened. Television cameras with recorders were placed at the viewing port in the dump or vent line and the viewing port in the vacuum tank. A recorder which handled the output of the pressure transducers was started, and the system was ready to run. The test was then initiated by opening the dump or vent valve, whichever was appropriate. The data gathered during a run consisted of the recorded output of the pressure transducers and the video tape of the viewing port observations.

SECTION V

RESULTS

The results of three of the runs will be presented in some detail. The other four runs will be discussed briefly since they will be quite understandable from the previous discussion contained in this report.

The pressure data acquired during Run A are shown in figure 4. The data show that the N_2O tank pressure was 1200 psia at the start of the run, but had fallen to 350 psia when the last bit of liquid was expelled. Apparently the liquid N_2O flowed out of the tank faster than the helium pressurant flowed in, after the dump valve was opened to start the run. This is also reflected in the pressure measured at the dump line outlet. This pressure was equal to the vacuum tank pressure (0.5 psia) at the start of the test. After the dump valve was opened, the dump line outlet pressure rose to 200 psia in 0.29 second. This is the starting transient. The pressure then diminished as the N_2O tank pressure diminished, reaching a low of 80 psia when the liquid N_2O was depleted. The minimum line pressure of 80 psia is well above the 12.7 psia required for solid formation, thus we would not expect any solid to be formed in the dump line.

The TV coverage of the viewing port at the dump line exit was inconclusive. It was not possible to determine what phases were flowing from these data, not only for Run A but for all of the runs. Based upon the pressure data, it was concluded that no solid formed in the line. The TV coverage of the viewing port in the vacuum tank showed that solid (snow) was present there. This would be expected since the pressure in the vacuum tank varied from 0.5 to 10 psia during the run, and thus remained below the triple point pressure of 12.7 psia. Note that the pressure drop across the dump line exit, in this case, was a minimum of 70 psi. Thus, no solid was formed in the dump line, but the expansion of the N_2O, when it left the dump line exit, was sufficient to form snow beyond the vent line exit. No problems were encountered in dumping the 24 pounds of N_2O.

The results of Run C are similar to those of Run A and the pressure data are plotted in figure 5. The N_2O tank pressures are lower since no helium was used, but the trends are the same. The minimum pressure at the dump line exit was 48 psia, which is considerably higher than the 12.7 psia required for solidification. Once again, solid N_2O was observed downstream from the dump line exit.

Runs B and D were similar to Runs A and C, except that these runs used only 12 instead of 24 pounds of N_2O. Since more gas was present in the tanks during Runs A and C, the N_2O tank pressure remained somewhat higher during these runs. As a result, the dump line exit pressures were also higher. No solid formed in the dump line, but snow was formed downstream of the dump line exit in both runs.

Gas was vented in Runs E and F. No solid was formed either in the vent line or downstream of the vent line exit. This is in accord with the theoretical analysis. It was found, however, that venting of the gas for periods of 3 or 4 minutes caused the liquid in the tank to solidify. The solid resembled snow rather than ice, and could be blown out of the tank by using helium as a pressurant.

Run G was a mixed mode test. The results are presented in figure 6. The purpose of this test was to dump 12 lbs of liquid with the dump beginning when the tank pressure corresponded to a temperature of less than 0°F. Recall that the N_2O temperature in the airborne system is maintained at 0°F (vapor pressure of about 250 psia). The system used for the test program maintained the N_2O at 32°F (vapor pressure of about 550 psia). It was deemed desirable to make one liquid dump run with the starting pressure below 250 psia. Figure 6 shows that venting the gas for 5.5 seconds caused the N_2O pressure to fall to 200 psia. After 5.5 seconds, the vent valve was closed and the dump valve was opened took 4.1 seconds to dump somewhat less than 12 pounds of liquid under these conditions. There was no solidification in the line since the line pressure never fell below 12.7 psia. Snow was present in the vacuum tank. No flow problems were encountered.

It may be of interest to look at the average flow rates achieved during the dump runs. These are shown in table 1, and all of the flow rates are in a correct relationship with each other, i.e., a higher tank pressure results in a higher flow rate.

SECTION VI

CONCLUSIONS

No solidification of N_2O occurred in the lines during gas venting or liquid dumping. The N_2O liquid did solidify after exiting from the line and entering the vacuum chamber, but this condition does not cause problems with the flow.

Excessive gas venting will cause freezing of liquid N_2O in the tank. No solidification occurs in the lines.

Table 1

FLOW RATES

Run No.	Mass of N_2O (lbs)	N^2O Tank Pressure (psia)	Time (sec)	Flow Rate (lb/sec)
A	24	1200-350	6.3	3.8
B	12	1200-850	1.5	8.0
C	24	600-100	7.4	3.2
D	12	550-350	3.7	3.2
G	>12	200-100	3.7	>3.2

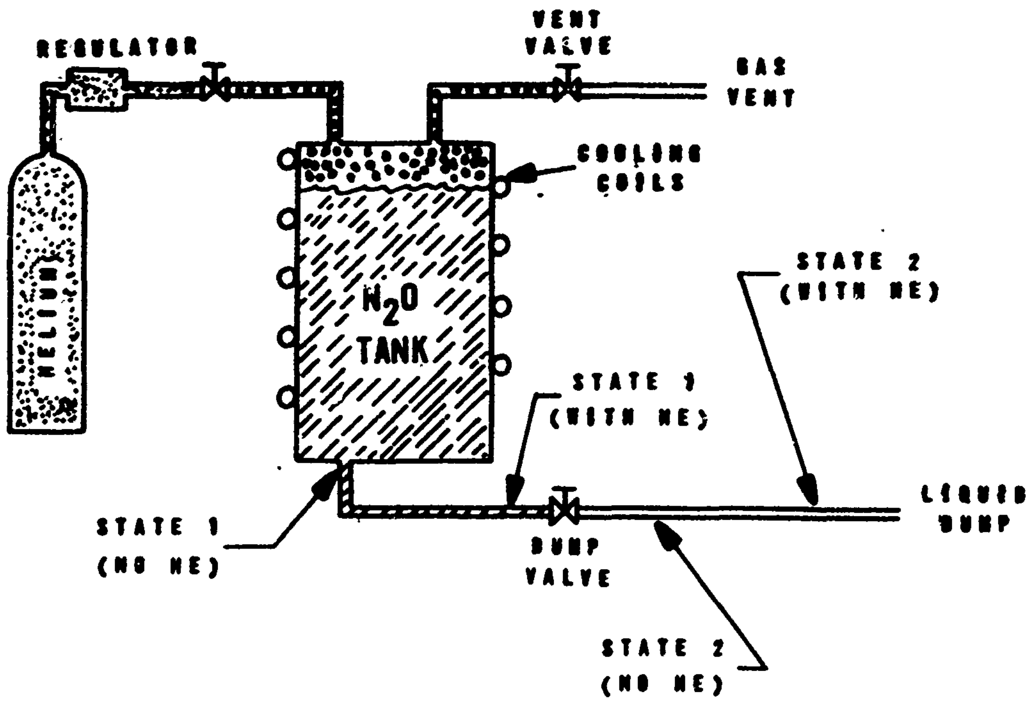

Figure 1. Schematic Diagram of N_2O System

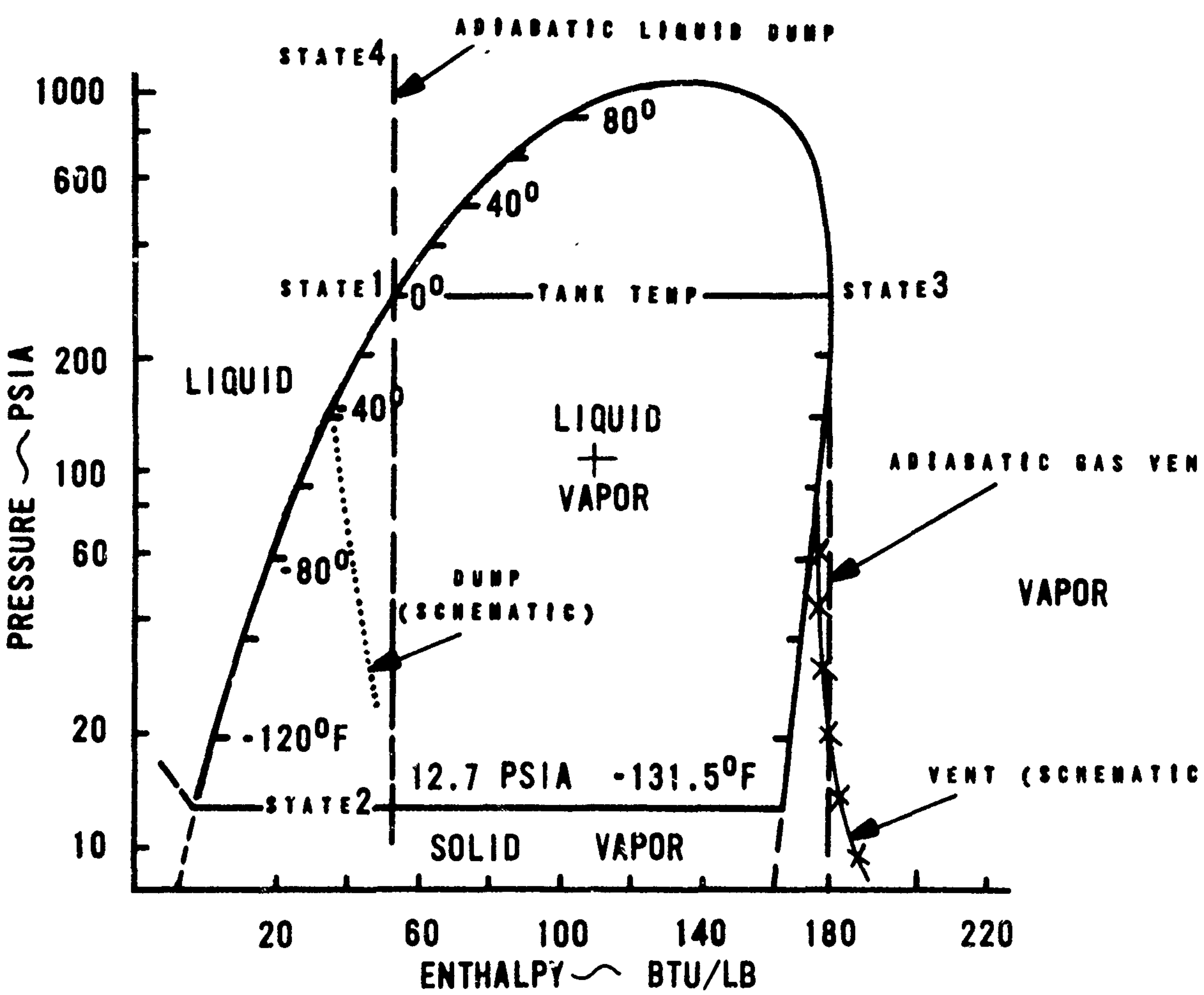

Figure 2. Pressure - Enthalpy Diagram of N_2O

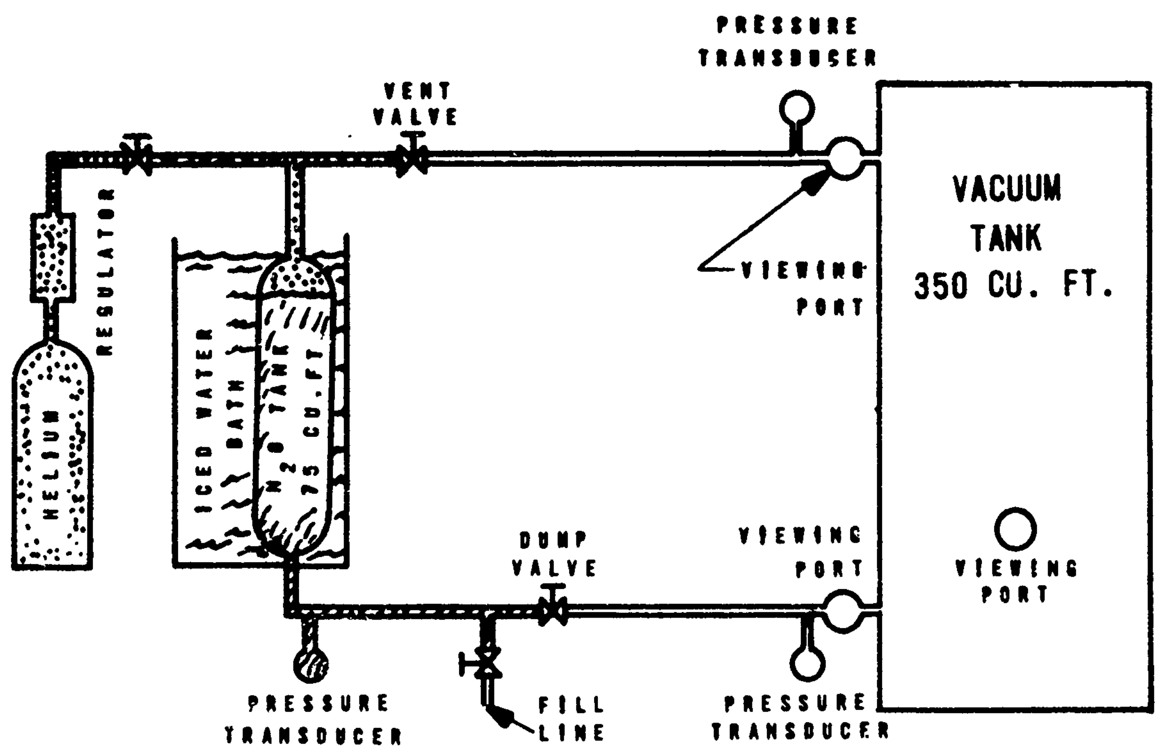

Figure 3. Test Facility Schematic

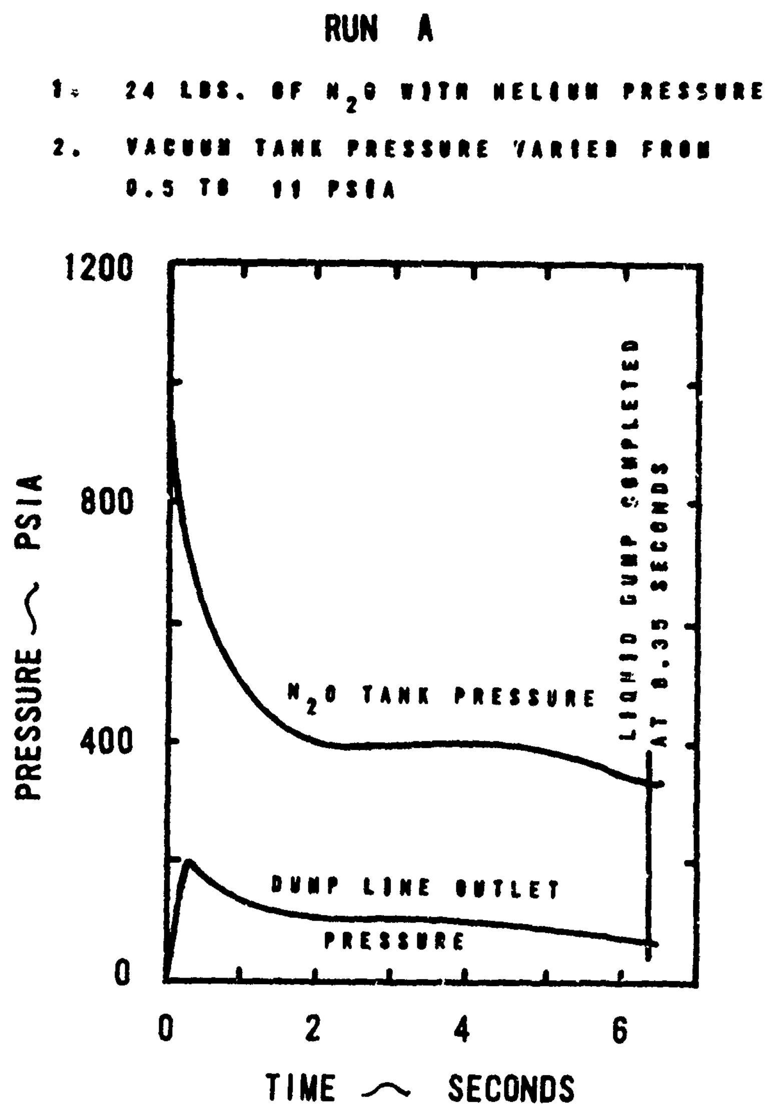

Figure 4. Pressure Data for Run A

RUN C

1. 24 LBS OF N_2O WITH NO HELIUM

2. VACUUM TANK PRESSURE VARIED FROM 0.5 TO 11 PSIA

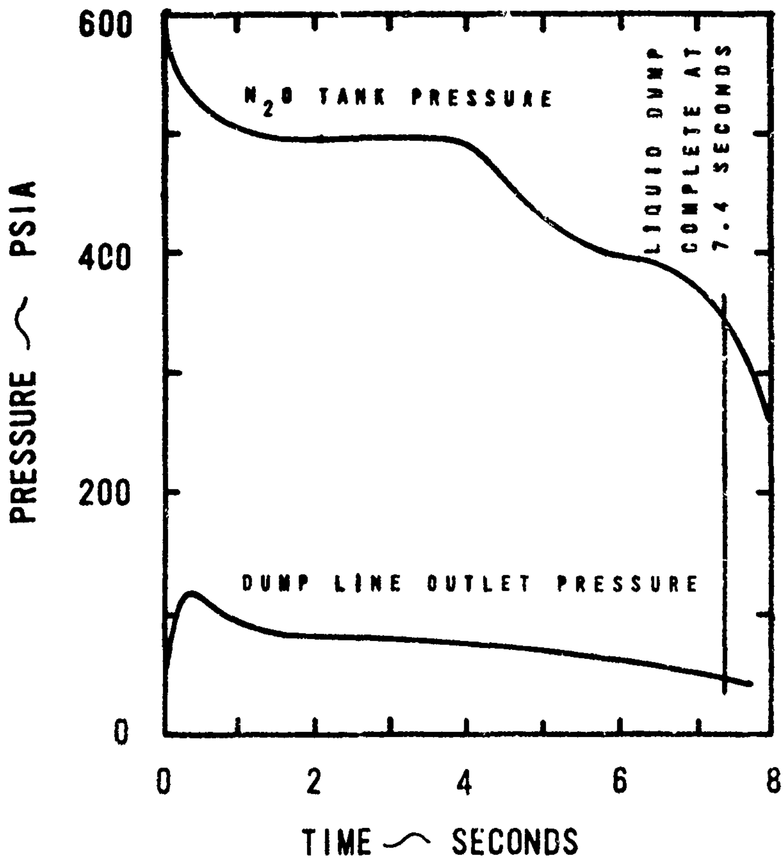

Figure 5. Pressure Data for Run C

RUN G

1. 12 LBS OF N_2O WITH NO HELIUM
2. VACUUM TANK PRESSURE VARIED FROM 0.5 TO 6 PSIA
3. GAS VENT FOR 5.5 SECONDS, THEN LIQUID DUMP UNTIL 9.2 SECONDS.

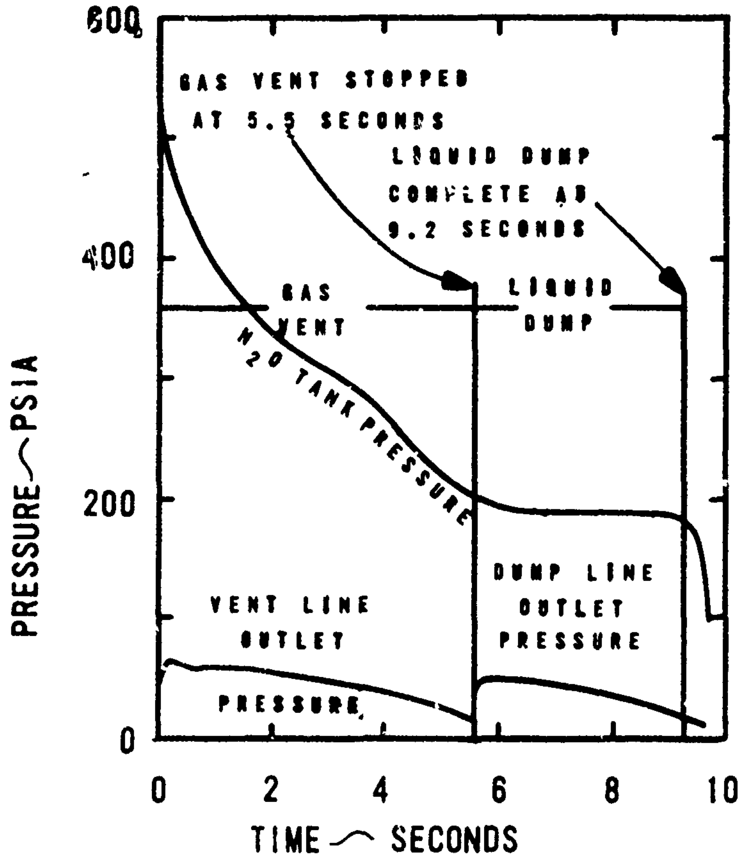

Figure 6. Pressure Data for Run G